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viscoelastic properties of aqueous  
polyethylene oxide solutions using an elastometer

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INVESTIGATION OF LOW FREQUENCY DYNAMIC  
VISCOELASTIC PROPERTIES OF AQUEOUS  
POLYETHYLENE OXIDE SOLUTIONS USING  
AN ELASTOMETER

JAMES CHARLES VOHR, JR.

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INVESTIGATION OF LOW FREQUENCY DYNAMIC  
VISCOELASTIC PROPERTIES OF AQUEOUS  
POLYETHYLENE OXIDE SOLUTIONS USING  
AN ELASTOMETER

by

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Lieutenant, United States Navy  
B. A., Dartmouth College, 1957

Submitted in partial fulfillment  
for the degree of

MASTER OF SCIENCE IN PHYSICS

from the

UNITED STATES NAVAL POSTGRADUATE SCHOOL  
May 1966



# ABSTRACT

The viscoelastic properties of dilute aqueous solutions of polyethylene oxide were studied using a modified simple elastometer. The dynamic viscosity and dynamic rigidity are calculated from the measurements in the frequency range from 1 to 11 Hertz for concentrations of .25%, .5%, 1%, and 1.5% of a polymer having a molecular weight of about 4 million.

The results indicate that the solutions may be represented as a simple Maxwellian element in the frequency range considered.

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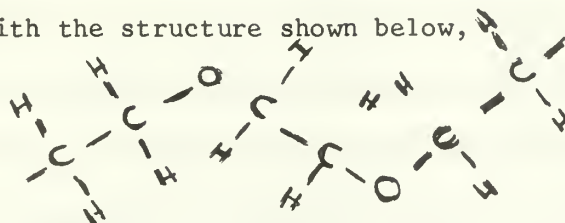
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## 1. Introduction

Polyethylene oxide, a high molecular weight, long-chain linear molecule with the structure shown below,



has been the subject of many recent investigations. When dissolved in water, it forms a viscoelastic solution which exhibits some of the characteristics of both a solid and a liquid. That is, it has an elastic modulus and a coefficient of viscosity. Polyethylene oxide solutions also exhibit a decrease in the apparent viscosity with increasing rates of shear, which is a characteristic of so called pseudo-plastic or non-Newtonian fluids. Philippoff first found this phenomenon for certain fluids; he also found that the viscosity of polymers under oscillating deformations depends only on the frequency and not on the amplitude of deformation. (7)

Non-Newtonian fluids are best characterized by a complex viscosity or impedance function  $\eta^* = \eta' - i\eta_2$ . Where  $\eta'$  is the usual dynamic viscosity and  $\eta_2 = \frac{G'}{\omega}$  where  $G'$  is the dynamic rigidity and  $\omega$  is the angular frequency of oscillation.

The real and imaginary parts of  $\eta^*$  give good insight into the properties of the solution under periodic deformation. If  $\eta_2$  is zero, the fluid is perfectly viscous, and if  $\eta'$  is zero, the fluid is perfectly elastic. The use of a complex impedance, however, is useful only for oscillatory motion.

Polyethylene oxide solutions have been the subject of two thesis studies at this institution. Lt. Chester found, in 1964, that these solutions behave essentially like water for sound waves at relatively

high frequencies (10 to 26 megaHertz). (1) The long-chain molecules in solution contribute no measureable effects to the absorption or speed of sound. He did find, however, that shear waves in the megaHertz range are strongly affected by the addition of the polymer. In 1965, Lt. Kinnier and Lt. Reister found, using a torsional oscillator, that from 118 Hertz to 675 Hertz, viscosity changes with frequency, and that the long-chain molecules have a great effect on the properties of the solution. (4)

The object of this investigation was to determine the viscoelastic properties of the solutions in the low oscillatory frequency range from 1 to 10 Hertz. The polyethylene oxide used in this investigation was purchased from the Union Carbide Company. It is named Polyox WSR-301; its average molecular weight is approximately  $4 \times 10^6$  and the distribution is unknown.

## 2. Equipment

In order to measure the complex viscosity at low frequency in Polyox, a modified simple elastometer was constructed, following the designs of Goldberg and Sandvik, (3) and Markovitz, Yavorsky, Harper, Zapas and DeWitt. (6) Goldberg and Sandvik designed and constructed a simple elastometer which provided a quick and simple method for determining viscoelastic properties of gels and soap solutions. Its operation depends on the motion of a cylindrical bob suspended in fluid undergoing periodic angular oscillation. Its operation, however, is limited to the frequency of natural resonance for the suspended cylinder. Markovitz, et al took the basic design of Goldberg and Sandvik and extended its frequency range.

The equipment used in this investigation is similar to the elastometer of Markovitz, but utilizes an optical method for obtaining the needed measurements. It consists essentially of a circular cylinder containing a liquid, in this case Polyox, in which another smaller cylinder is suspended concentrically. The outer cylinder is made to oscillate mechanically through a small angle with a frequency that can be varied. The motion of the inner cylinder with respect to the outer cylinder can be used to determine the complex viscosity of the fluid.

The apparatus (Figures 1 and 2) consists of a steel table with three leveling legs, on which a turntable with a lathe-turned shaft is mounted. The turntable is supported by ball bearings and is rigidly connected to a light aluminum rocker arm. A smooth rubber-covered wheel mounted at the end of the rocker arm is held by a spring against an eccentric cam. The eccentric cam is driven by a shaft and pulley system coupled to a variable speed electric motor. Since the drive



motor is more stable in operation at high speeds, two sets of pulleys are used to reduce the shaft speed. The pulley system also isolates the steel table from any motor vibration.

If the eccentricity of the cam is small compared to the length of the rocker arm, then the motion of the turntable can be considered sinusoidal.

A metal can is mounted on the turntable with a smooth glass cylinder cut from a length of large diameter glass tubing fixed in the center. This glass tube serves as the outer cylinder and the metal can as a container for a constant temperature bath.

Several different diameter inner cylinders were constructed using machined and polished Lucite rod. The inner cylinder is suspended by a thin (No. 38) copper wire fixed to a screwjack device which permits the suspension height to be varied. The screwjack device can also be moved laterally to permit centering of the inner cylinder.

Optical means are employed to measure the motion of the inner and outer cylinders. This system functions as follows: a mirror is mounted on the side of the outer cylinder and another mirror is mounted on the inner cylinder. These two mirrors are illuminated with a highly collimated beam of light from a common light source. The reflected beams of light are displayed on a curved screen placed three meters from the inner cylinder. The curved screen eliminates any distortion of the end points of the arcs of reflected light. Although the reflecting mirrors are not the same distance from the screen, the distance of separation is small compared to the distance to the screen so that the error caused by this is negligible.

Two photocells are mounted behind slits on the curved screen.

They can be positioned in the midpoint of the arc of reflected light from the oscillating cylinders. The output of each one of these cells is amplified and displayed on separate traces of a dual beam cathode ray oscilloscope (Figure 3). This permits measurement of the phase angle between the displacements of the inner and outer cylinder by using a vernier delay dial to align the output traces. The amplitude of the oscillation is measured directly on the face of the screen by measuring the length of the light arc.

There was a slight amplitude modulation of the magnitude of both reflected arcs. This may have been caused by a minor wobble in the turntable, resulting from the light load on the supporting thrust bearings.

This apparatus is useful in the frequency range from 1 to 11 Hertz. At higher frequencies the rocker arm tends to float or vibrate on the eccentric cam, and at lower frequencies, the size of the inner cylinder, the restoring force of the wire, and their relation to the viscosity of the fluid becomes very critical.

### 3. Theory of Measurement

The measuring device (Fig. 4) consists of a lucite cylinder of radius  $r$  suspended to depth  $h$  in a liquid contained in another cylinder of radius  $R$ . The outer cylinder is caused to execute simple harmonic oscillations through a small fixed angular amplitude. The relationship between the motion of the inner cylinder and the viscoelastic properties of the liquid was developed by Markovitz. (5,6)

Consider a cylinder lamina of the liquid of height  $h$  with inner radius  $z$  and outer radius  $z + dz$ . The torque which acts on the cylindrical surface of the cylindrical lamina at  $z$  is  $-2\pi z^2 \eta h \frac{\partial \dot{\Theta}}{\partial z}$ , where  $\dot{\Theta}$  is the angular velocity of the liquid at  $z$  and  $\eta$  is the viscosity coefficient, which may be either real or complex. At  $z + dz$ , the torque on the surface is  $2\pi z^3 \eta h \frac{\partial \dot{\Theta}}{\partial z} + \left\{ 2\pi z^3 \eta h \left( \frac{\partial \dot{\Theta}}{\partial z} \right) \right\} dz$ , and the net torque on the lamina is

$$\left\{ 2\pi z^3 \eta h \left( \frac{\partial \dot{\Theta}}{\partial z} \right) \right\} dz$$

As a result, the element of volume will acquire an angular acceleration  $\ddot{\Theta}$  such that  $2\pi z^3 \rho \ddot{\Theta} dz = \left\{ 2\pi z^3 \eta h \left( \frac{\partial \dot{\Theta}}{\partial z} \right) \right\} dz$ . Since the motion is a forced oscillation with frequency  $\frac{\omega}{2\pi}$  the substitution  $\Theta = (\theta) e^{j\omega t}$  can be made, and the equation of motion becomes

$$\frac{d^2 \theta}{dz^2} + \left( \frac{3}{z} \right) \frac{d\theta}{dz} - j \frac{\rho \omega \theta}{\eta} = 0$$

The solution of this equation is  $\Theta = \frac{[A J_1(\alpha z) + B Y_1(\alpha z)]}{z}$  where  $J$  and  $Y$  are Bessel functions of the first order,  $A$  and  $B$  are arbitrary constants to be determined by the boundary conditions and  $\alpha$  is  $\left( -j \frac{\rho \omega}{\eta} \right)^{1/2}$ .

. At the inside wall of the cylinder containing the liquid, the motion can be represented by  $\Theta(R) = \Theta_R e^{j\omega t}$  since the liquid is

following the forced motion of the cylinder which has amplitude  $\Theta_R$  the suspended lucite cylinder experiences a shearing torque equal to  $2\pi r^3 h \eta \left( \frac{\partial \Theta}{\partial z} \right)_r$  due to the liquid and a restoring torque  $-K\Theta_r$  due to the suspension wire which has a torsional constant  $K$ . As a result the bob acquires an angular acceleration such that

$$2\pi h \eta r^3 \left( \frac{\partial \Theta}{\partial z} \right)_r - K\Theta_r = I \ddot{\Theta}_r$$

where  $I$  is the moment of inertia of the cylinder. Putting the boundary conditions into the solution for  $\Theta$  one can arrive at the equation of motion for the inner cylinder.

$$\ddot{\Theta}_r = \frac{a^2 \Theta_R r^2 [J_1'(ar) Y_1(ar) - J_1(ar) Y_1'(ar)]}{(1 + br^2) [Y_1(ar) J_1'(aR) - J_1(ar) Y_1'(aR)] - abR^2 [J_1'(aR) Y_1(aR) - J_1(aR) Y_1'(aR)]}$$

where  $b = \frac{-2\pi h \eta \omega}{(I\omega^2 - K)}$  and  $J_1'(aR)$  is the value of  $\frac{dJ_1(aR)}{d(aR)}$  at  $z=R$

Although this represents the exact relationship between the desired quantity  $\eta$  and the measured quantities  $\Theta_r$  or  $\ddot{\Theta}_r$  it is in an inconvenient form since  $\eta$ , the unknown, is buried in the complex argument of the Bessel functions. Making a Taylor expansion of the functions with argument  $ar$  about  $aR$  the following result is obtained:

$$\begin{aligned}
\frac{\Theta_R}{\Theta_r} = & 1 + \frac{i}{n} \left[ \frac{I\omega^2 - K}{4\pi h \omega} \frac{R^2 - r^2}{R^2 r^2} + \frac{\omega \rho}{8} \frac{(R^2 - r^2)^2}{R^2} \right] \\
& - \frac{i}{n^2} \left[ \frac{(I\omega^2 - K)\rho}{32\pi h} \left( 4 \ln\left(\frac{r}{R}\right) + \frac{R^2}{r^2} - \frac{r^2}{R^2} \right) \right. \\
& \quad \left. + \frac{\omega^2 \rho^2}{192 R^2} \left\{ (R^2 - r^2)(R^4 - 5R^2 r^2 - 2r^4) + 12R^2 r^4 \ln\frac{R}{r} \right\} \right] \\
& + \frac{i}{n^3} \left[ \begin{array}{cccc} - & - & - & - \end{array} \right] \\
& + \begin{array}{cccc} - & - & - & - \end{array}
\end{aligned}$$

Letting  $\Theta_R = |\Theta_R| e^{j\omega t}$      $\Theta_r = |\Theta_r| e^{j[\omega t - \phi]}$

$$m = \frac{|\Theta_r|}{|\Theta_R|}$$

$$A_1 = \frac{I(R^2 - r^2)}{4\pi h R^2 r^2} \quad B_1 = \frac{(R^2 - r^2)^2}{8 R^2} \quad C_1 = \frac{K A_1}{I}$$

$$A_2 = \frac{I}{32\pi h} \left[ 4 \ln \frac{r}{R} + \frac{R^2}{r^2} - \frac{r^2}{R^2} \right]$$

$$B_2 = \frac{1}{192 R^2} \left[ (R^2 - r^2)(R^4 - 5R^2 r^2 - 2r^4) + 12 R^2 r^4 \ln \frac{R}{r} \right]$$

$$C_2 = \frac{K A_2}{I}$$

and neglecting higher order terms results in

$$\frac{\Theta R}{\Theta r} = \frac{|\Theta R|}{|\Theta r|} \frac{e^{j\omega t}}{e^{j(\omega t - \phi)}} = \frac{1}{m} e^{j\phi} = \frac{1}{m} [\cos\phi + j\sin\phi]$$

$$\frac{1}{m} [\cos\phi + j\sin\phi] = 1 + \frac{j}{n} [(A_1 + B_1\rho)\omega - \frac{C}{\omega}] - \frac{1}{n^2} [(A_2 + B_2)\rho\omega^2 - C\rho]$$

$\mathcal{R}$  now may be found by the solution of a complex quadratic equation or, if the term involving  $\frac{1}{n^2}$  may be neglected, then by a first order equation.

$$1 - \frac{1}{m} \cos\phi - \frac{j}{m} \sin\phi + \frac{j}{n} [(A_1 + B_1\rho)\omega - \frac{C}{\omega}] = 0$$

In this investigation it was possible to neglect all terms of higher order than  $\frac{1}{n}$ , because of the errors in the measurement of phase angle and amplitude.

All of the constants may be calculated from direct measurements except for the torsional constant of the suspension wire. The torsional constant may be determined from the frequency of oscillation of the inner cylinder suspended in air using the relation

$$k = (2\pi f)^2 I$$

The values of the constants for the three inner cylinders used are shown in Table 1.



#### 4. Experimental Procedures

Basically, the operation consisted of filling the inner cylinder with the sample of Polyox to be tested, then immersing and centering the inner cylinder. The relative magnitude of oscillation and phase lag angle is then recorded as a function of oscillation frequency.

The samples of Polyox were all mixed in a 2000 ML beaker with a three bladed beater at 100 RPM. The powdered Polyox was slowly added during the mixing cycle and care was taken not to form clumps in the solution.

The turntable was carefully leveled before a run to insure that the inner cylinder would be suspended concentrically with the outer cylinder.

The inner cylinder was centered before the Polyox was introduced by checking its position over a pointed plug which fitted in the outer cylinder center. The inner cylinder then was lowered to a prescribed depth after the outer cylinder had been filled.

The response of the elastometer is very dependent on the viscosity of the fluid, the distance of separation between the cylinders, and the frequency of oscillation. As an example, in a 1% solution of Polyox with a 12 mm. diameter inner cylinder and a 14.4 mm. diameter outer cylinder at frequencies of less than 3 Hertz, both cylinders appear to move together, and at frequencies higher than 8 Hertz, the motion of the inner cylinder becomes very small. Accurate measurements of viscosity are difficult under either of these conditions. The radius of the inner cylinder, or the restoring force, must be varied in order to cover the frequency range of interest. In this investigation, different inner cylinders were used with the same outer cylinder.

Most of the problems encountered were mechanical in nature, such as the centering of the inner cylinder, and the stability of motor operation.

Although the apparatus was constructed with a constant temperature jacket, this was not employed during the runs. The measurements were made at room temperature, which was  $24 \pm 1^{\circ}\text{C}$ . The dynamic viscosity of Polyox is temperature dependent but Chester's results indicate that any uncertainty caused by a  $\pm 1^{\circ}$  temperature variation would be less than the experimental error.



## 5. Results and Discussion

The results are best shown graphically (Figures 4,5) with the plots of  $\eta'$  the dynamic viscosity, and  $G'$  the modulus of rigidity, vs. frequency for different concentrations of Polyox. Selected values are also presented in Table 2. These two quantities are plotted with a straight line approximation for different concentrations. The dynamic viscosity decreases with increasing frequency of oscillation while the modulus of rigidity increases.

$\eta'$  and  $G'$  are also plotted (Figure 6) as a function of concentration at a frequency of 3 Hertz. Both  $\eta'$  and  $G'$  change greatly with increasing concentration and both of the plots have the same general shape.

An exact estimation of the amount of error is difficult to make in this investigation. The elastometer was tested with a Newtonian solution of known viscosity and the results checked within 10%. The error appeared to be random, so it could not be used as a corrective factor to be applied to other measurements.

The most critical variable was the phase lag angle. Since  $\eta'$  and  $G'$  are direct functions in the computation formula of the sine and cosine of the phase lag angle, they were markedly affected by any errors in that angle. This was particularly true when the angle measured was near  $0, \frac{\pi}{2}$  or  $\pi$  where the sine or cosine change most rapidly.

The phase angle is determined by measuring the ratio of the sweep delay to the period. For small delays the uncertainty in measurement due to lack of trace stability and eye estimation is of the same order of magnitude as the observed quantity. An estimate of the uncertainty in the phase angle is  $\pm 3^\circ$ .

A phase angle meter was also used to try to improve the accuracy of the measurement; however, it was not capable of giving good results on the irregular pulse outputs from the photocells.

The amplitude ratio was easily measured to within an accuracy of  $\pm 5\%$  since both light beams swung through an arc ranging from  $1\frac{1}{2}$  to 8 inches in length.

In a given series of measurements with a particular inner cylinder, the amplitude ratio starts at unity for low frequencies, increases to a maximum at system resonance, then decreases as frequency increases. The phase lag angle starts at zero, increases to  $\frac{\pi}{2}$  at resonance, and approaches  $\pi$  as the frequency increases. A value of  $\eta'$  and  $G'$  was determined at whatever frequency the amplitude ratio and phase lag angle was measured, and these values were used to plot the dependence on frequency. An alternate method would be to plot the phase lag angle and amplitude ratio as a function of frequency and use values determined from this plot to calculate  $\eta'$  and  $G'$ . Regardless of the method used, each point on the plot would have a different estimate of error due to uncertainties in determination of frequency, of phase lag, of amplitude ratio, and of the other physical constants of the system. The points with the largest estimated error are the end points in a particular series of measurements, i.e., those at the low and high frequencies. The phase lag angle for these points is near 0 and  $\pi$  and any uncertainty in it affects the results greatly.

The straight lines were plotted by judgment and an overall error of  $\pm 10\%$  is considered reasonable, based on the results of measurements of fluids of known viscosity.

While the investigation was in progress, it was learned that

Michael C. Williams at the University of Wisconsin had made similar measurements on 1.46% Polyox solution using a Birnboim concentric cylindrical pumping viscometer. His measurements covered a wider frequency band but his results match the results of this investigation and those of Lt. Kinner and Lt. Reister very closely. His results are plotted with the results of this investigation.

The simplest way of accounting for the mechanical properties of Polyox solutions is to introduce a single viscosity  $\eta_0$  and a single shear modulus  $G_0$ . One method of superimposing these properties is the Maxwell model. In the Maxwell model, the fluid is represented by a Maxwellian element consisting of a spring (representing the shear modulus) and a dashpot (representing the viscosity) in series. If  $\eta_0$  is the zero shear rate viscosity,  $\omega$  is the frequency of the applied disturbance, then the Maxwell model for viscoelastic solutions gives a formula for the dynamic viscosity

$$\eta' = \frac{\eta_0}{1 + \omega^2 \tau^2}$$

where

$$\tau = \frac{2\pi\eta_0}{G_0}$$

$G_0$  = the shear modulus at infinite shear.

Using values of  $\eta'$  determined in this investigation at 3 Hertz and 6 Hertz to solve the equation of the simple Maxwell model for  $\eta_0$  and  $\tau$  for various concentrations of Polyox, values for  $\eta_0$  are computed which are the same order of magnitude as the low shear rate viscosity coefficients measured at 6 RPM with a Brookfield viscometer. The values are compared with the relaxation times in Table 3. The agreement between  $\eta_0$  is better at the lower concentrations and these results indicate that in the frequency range from 1 to 11 Hertz the

behavior of Polyox solutions may be approximated by a single Maxwell element. The results of Lt. Kinnier and Lt. Reister indicate that as frequency increases, this approximation is no longer valid.

The elastometer did not prove to be a good experimental device for investigating Polyox solutions. The properties of Polyox solutions change greatly with frequency and this requires many combinations of inner and outer cylinders to cover a limited frequency range. This investigation, I feel, has filled a gap in the Polyox spectrum. Further work is needed in smaller concentrations and at higher frequencies.

## 6. Acknowledgements

This research was conducted at the U.S. Naval Postgraduate School, Monterey, California. The writer wishes to thank Professor O.B. Wilson, Jr., for his intellectual support and H.D. Whitfill for his technical assistance.

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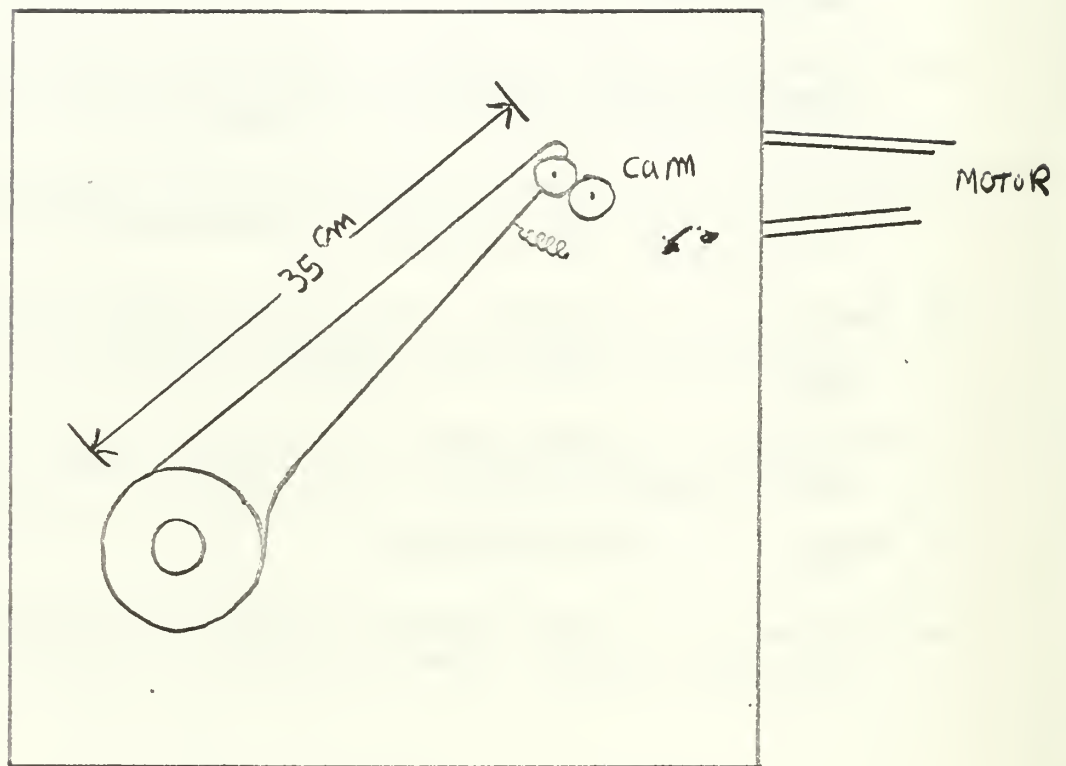


Figure 1. Top View of Elastometer

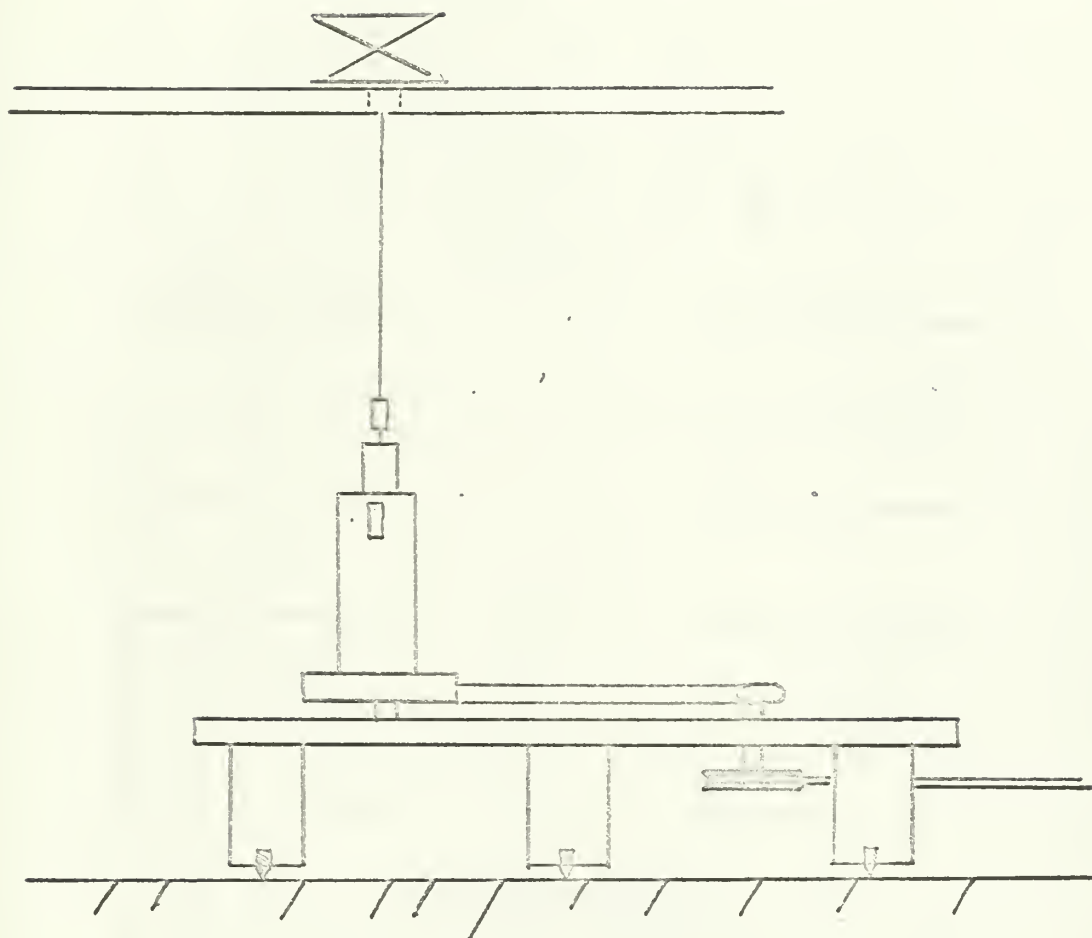


Figure 2. Side View of Elastometer



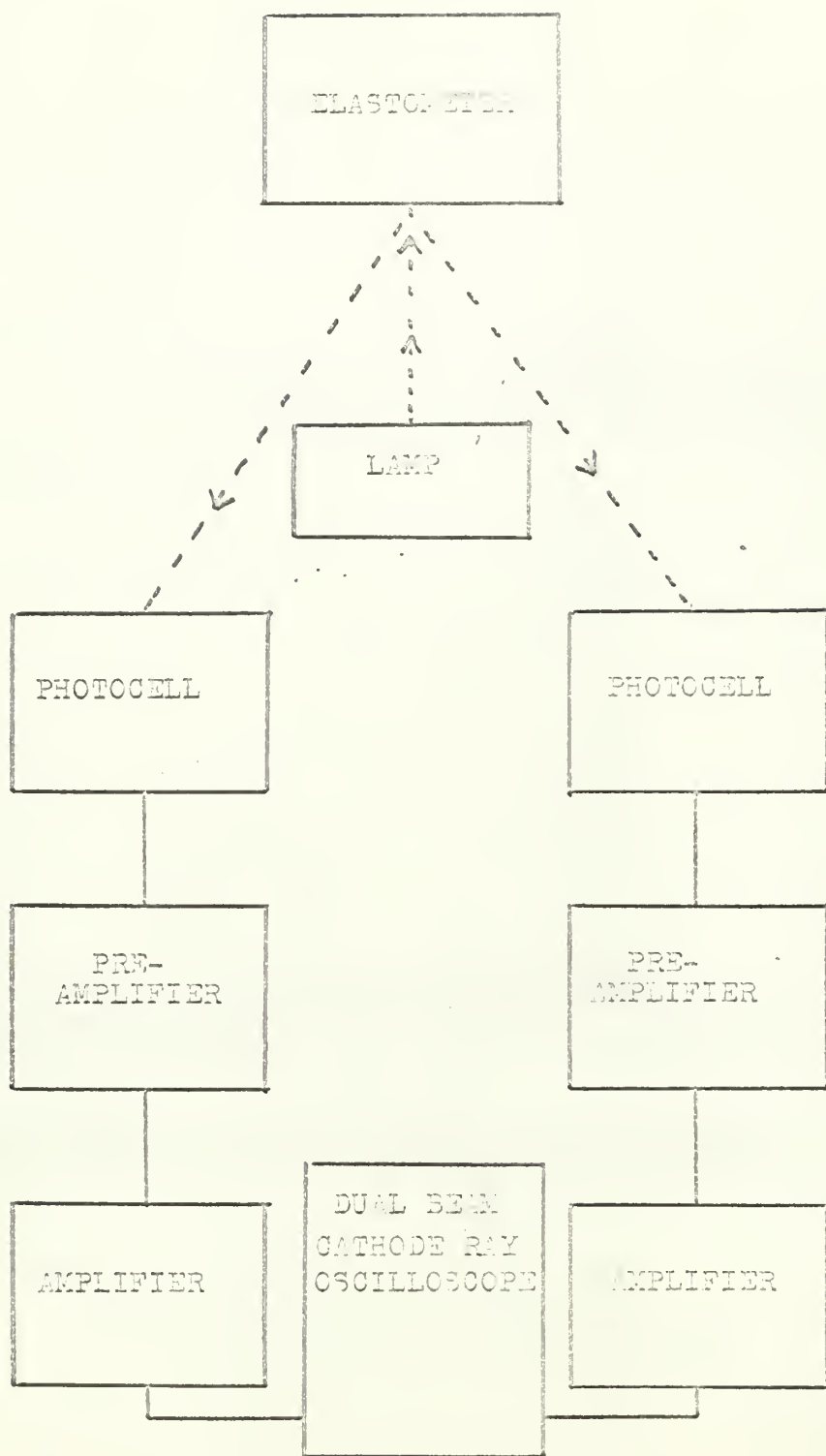


Figure 3. Block diagram of Measuring Apparatus

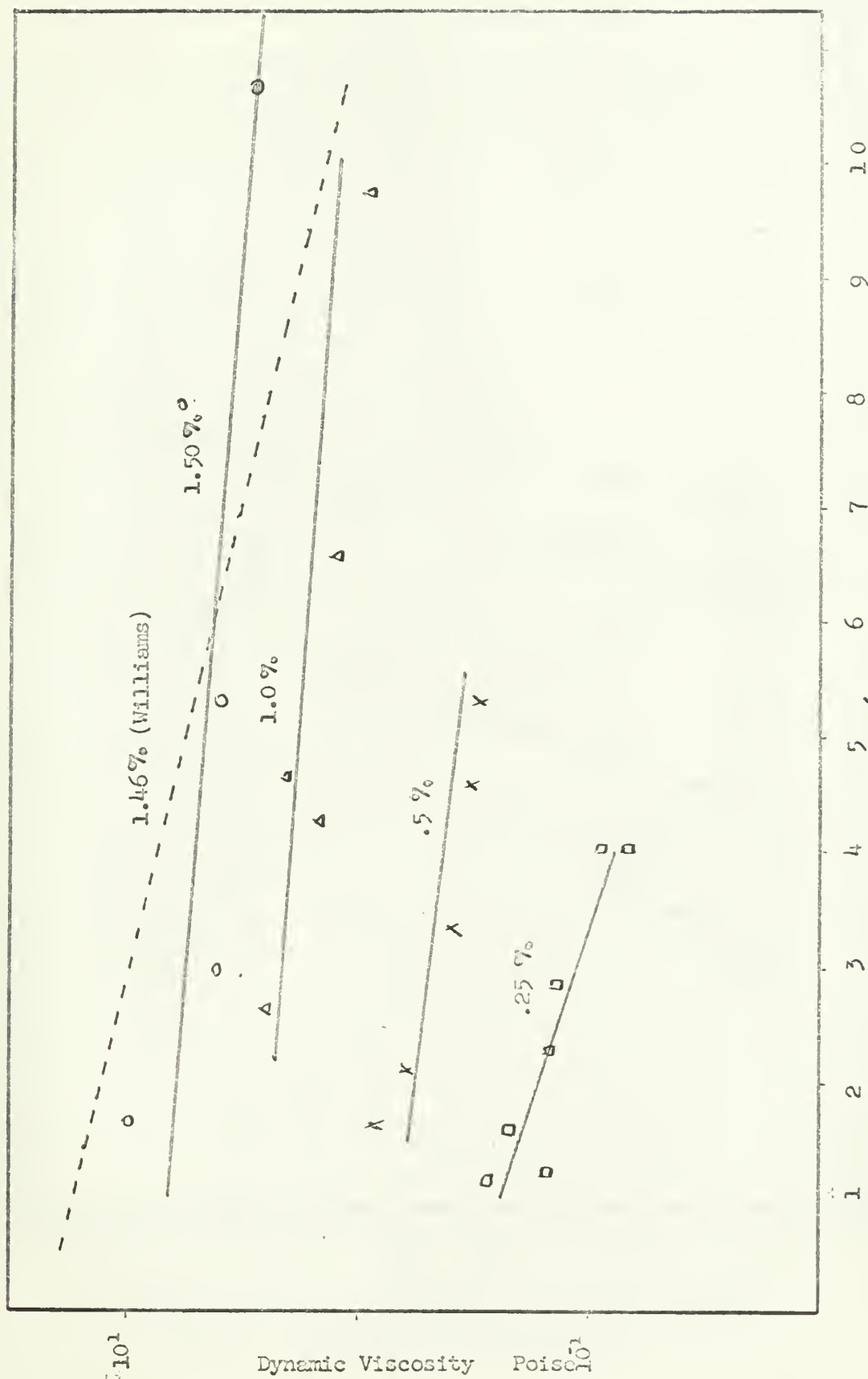


Figure 4. Dynamic Viscosity  $\eta'$ , versus Oscillation Frequency (Hertz) for Various POLYOX Concentrations.

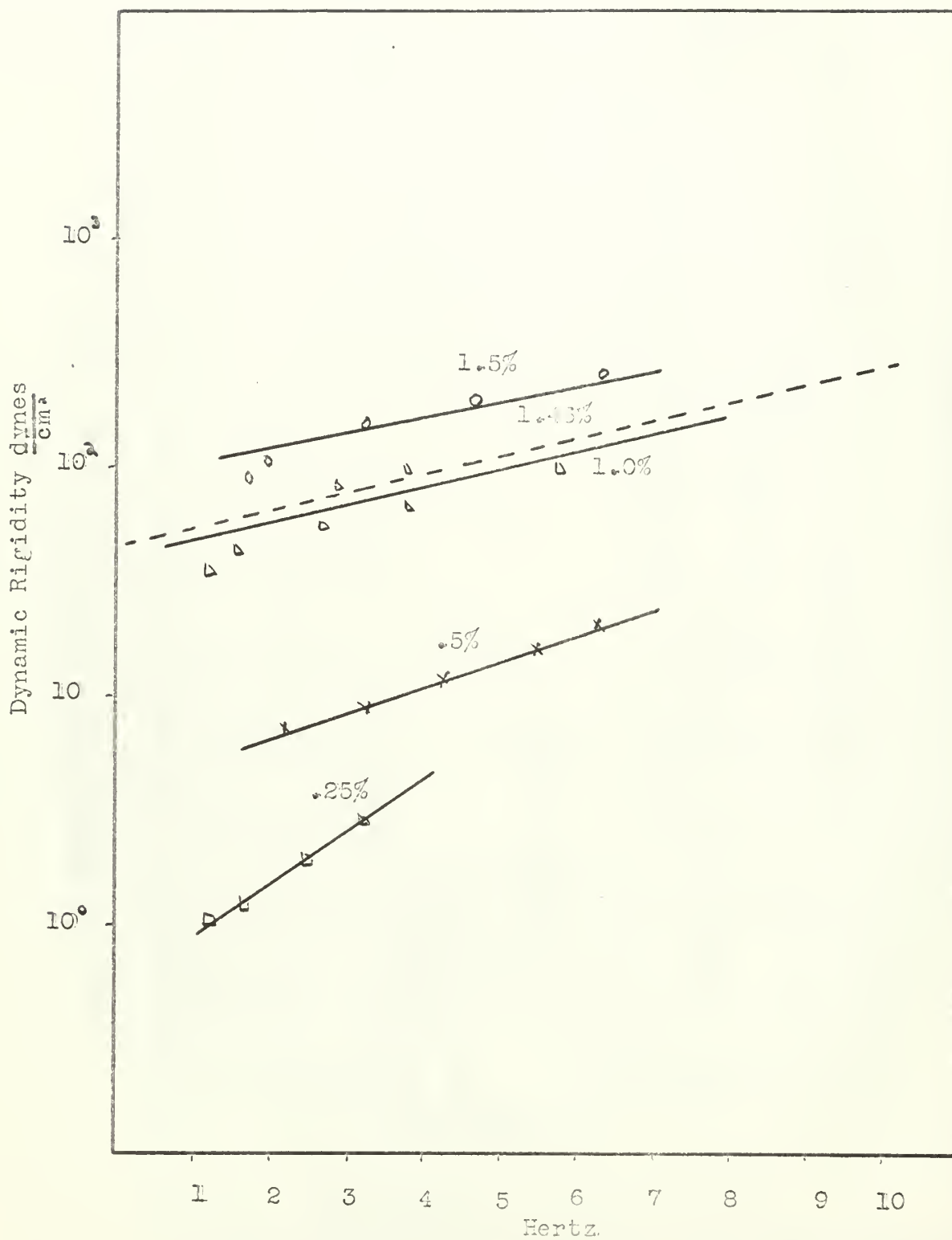


Figure 5. Dynamic Rigidity versus Frequency for various Concentrations of POLYCK

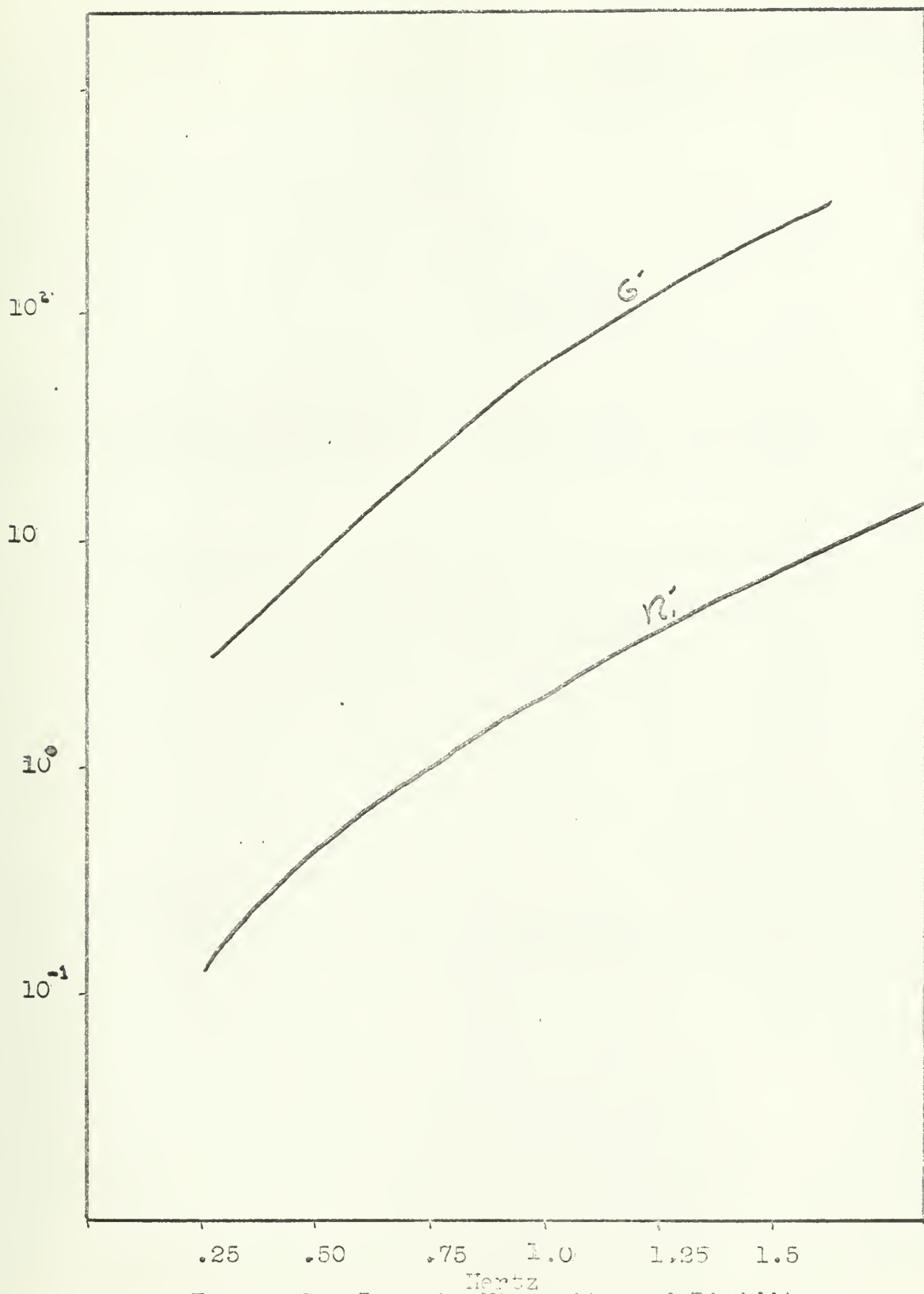


Figure 6. Dynamic Viscosity and Rigidity versus Concentration



Figure 7. Concentric Cylinders for Theory of Measurement

INNER CYLINDER RADIUS CM		.72	.6	.5
I	gm/cm	3.11	4.61	5.48
A <sub>1</sub>	g - cm	.0421	.0574	.110
B <sub>1</sub>	cm	.0177	.0428	.063
C <sub>1</sub>	gm/cm-sec	1.98	4.22	6.78

Table 1. Constants of the Elastometer

FREQUENCY HERTZ	CONCENTRATION PER CENT	$\eta'$ POISE	$G'$ DYNES CM
3	.25	.12	2.8
	.5	.5	8.6
	1.0	2.16	74
	1.5	5.60	171
	Willis 1.45	5.3	80
6	.25	--	--
	.50	.36	20
	1.0	1.7	134
	1.5	4.0	200
	Willis 1.46	4.0	136

Table 2 Derived Viscosity Coefficients

CONCENTRATION PER CENT	$\eta$ . CALCULATED POISE	$\tau$ MILLISECS.	BROOKFIELD 6 R.P.M. POISE
.25	--	--	.20
.50	.57	20.4	1.58
1.0	2.36	16.5	14.3
1.5	6.4	20.7	36

Table 3. Low Shear Viscosity Coefficients



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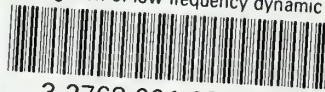






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